



Docket No. 4296-144 US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of	:	Customer No.
HIRAO et al.	:	26817
	:	
Serial No. 09/919,024	:	Group Art Unit: 1625
	:	
Filed: July 31, 2001	:	Examiner: OH, Taylor Victor
	:	
Title: METHOD FOR STARTING UP	:	Confirmation No. 3715
REACTOR	:	
	:	
	:	x

Mail Stop AF
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION OF HARUNORI HIRAO
SUBMITTED UNDER 37 CFR 1.132

Sir:

I, Harunori Hirao, hereby declare as follows:

1. I have a Master degree from Nagoya University.
2. I have performed experiments on a reactor for the reaction of catalytic gas phase oxidation using an apparatus for the production of acrylic acid illustrated in Fig. 2 of the above-described application which was used to perform Example 1 and Comparative Example 1 described on page 21, line 31 to page 23 line 19 and page 25, line 5 to page 26, line 5. Under the following conditions, a raw material to be oxidized, air, and a discharged gas from an absorption column were recycled to the reactor to initiate a reaction of catalytic gas phase oxidation therein. The reactor was a shell-and-tube type reactor made of steel and measuring 25.0 mm in inside diameter and 29.0 mm in outside diameter and furnished with 11500 reaction tubes. The shell of the reactor was a cylinder 4400 mm in inside diameter. Each of the reaction tubes was packed with 1520 cc of a reaction catalyst.
3. The reactor was set under target operating conditions, of a concentration, 4.5 vol. %, of propylene and a concentration, 10.0 vol. %, of oxygen at the inlet to the reactor and then the reactor was started up. It was found that the measurement made prior to the start of

the reaction, the concentration of propylene at the lower explosion limit was found to be 2 vol. % and the limiting oxygen concentration is 10.2 vol. % (in the propylene-oxygen-nitrogen system) at the temperature under the pressure both set in advance. The purity of the propylene supplied to the reactor was 99.5 vol. %.

4. The concentration of propylene at the inlet to the reactor was gradually increased, A: 0 vol. %, B: 2 vol. %, and C: 3 vol. %, by controlling the flow rate of air, steam, propylene, and discharged recycle gas through their respective lines with flow meters and flow control valves attached to the respective lines and the flow rate of air, discharged recycle gas, steam, and propylene and the concentration of oxygen at the inlet to the. The amounts of steam consumed till the points were reached were also measured. The results are shown in Table 1. In this table, the flow rate of air, discharged recycle gas, steam, and propylene and the total flow rate of gas were reported in the denomination of Nm^3/min , the concentration of oxygen at the inlet to the reactor was reported in vol. %, and the amount of steam consumed was reported in Kg.

[Table 1]

	Flow rate of air * (11)	Flow rate of discharged recycle gas (12)	Flow rate of steam (13)	Flow rate of propylene (14)	Total flow rate of gas (15)	Concentration of oxygen at inlet to reactor	Amount of steam consumed
A	303.0	204.1	35.0	0.0	338.0	16.2	845
B	296.3	206.9	35.0	6.7	338.0	10.0	1222
C	293.0	201.8	35.0	10.0	338.0	6.9	1509

*: The "Flow rate of air" includes the flow rate of the discharged recycle gas.

5. The amount of steam consumed was found to be about 2.5 tons and the duration of starting up the reactor to be about 2.5 hours.

6. The reactor was started up in the above-described manner, with the concentration of oxygen kept at less than the limiting oxygen concentration. The results are shown in Table 2.

[Table 2]

	Flow rate of air * (11)	Flow rate of discharged recycle gas (12)	Flow rate of steam (13)	Flow rate of propylene (14)	Total flow rate of gas (15)	Concentration of oxygen at inlet to reactor	Amount of steam consumed
A	183.0	150.0	155.0	0.0	338.0	9.5	3741
B	248.3	150.0	83.0	6.7	338.0	9.7	17526
C	268.1	150.0	59.8	10.1	338.0	9.8	20191

*: The "Flow rate of air" includes the flow rate of the discharged recycle gas.

7. The amount of steam consumed was found to be about 23.1 tons and the duration of starting up the reactor to be about 7 hours.

8. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and, further, that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Dated: Sep. 4. 2007 By: Harunori Hirao